

**Lee, Jang-Jung, Charles Evans & Associates Time-Of-  
Flight Secondary Ion Mass Spectroscopy (TOF-SIMS)  
Surface Analysis Report, CE&A Number 40150,  
March 18, 1994**

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# CHARLES EVANS & ASSOCIATES

Time of Flight-Secondary Ion Mass Spectroscopy Report

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## Company's Summary of the Charles Evans Results

Lehigh University has conducted an extensive investigation of the cathodes from heat producing as well as those from control cells and has determined that the results lend some support to Mills' theory. See Exhibit 6- Lehigh University XPS Report.

The XPS results were confirmed at National Laboratory A followed by Time of Flight-Secondary Ion Mass Spectroscopy (TOF-SIMS) analysis of the nickel surface. Iron and lithium were the only remaining atoms which were in question by Lehigh University and National Laboratory A as the source of the 54.6 eV XPS peak. The Charles Evans TOF-SIMS results demonstrate that iron and lithium were not the source of this peak. TOF-SIMS is orders of magnitude more sensitive in the identification of the presence of a given atom as compared to XPS. TOF-SIMS of Samples #1, #2, #3, #4 showed that no lithium was detected. TOF-SIMS of Sample 1# and Sample #2 showed that iron was at the detection limit which was far less than the amount required for an observable signal by XPS. TOF-SIMS of Sample #3, the control virgin nickel tube, contained six times the iron as Sample #4, but no 54.6 eV XPS peak was observed in the case of Sample #3. Therefore, iron was eliminated as the source of the 54.6 eV XPS peak of Sample #4. See Exhibit 7-Charles Evans Time of Flight-Secondary Ion Mass Spectroscopy Report.

The descriptions of the samples sent to Charles Evans & Associates by National Laboratory A are as follows:

### Sample #1

This sample is Sample # 9 of Lehigh University shown in Figure 34 of the Lehigh University XPS Report which is attached. The XPS was also performed on this sample at National Laboratory A. The survey spectrum is shown as Nitest21 which is attached. The spectrum in the region of 55 eV is shown as Nitest20 which is attached.

### Sample #2

Electrolysis followed by XPS was performed at National Laboratory A. The cathode comprised approximately 60 cm of 0.38 mm diameter nickel wire (99 % Alfa # 10249, cold drawn, clean Ni wire) that was cleaned by placing it in a beaker of 0.57 M  $K_2CO_3$  /3%  $H_2O_2$  for 10 hours and then rinsing them with distilled water. The anode comprised a 5 cm by 5 cm platinized titanium mesh. The electrolyte solution was 0.57 M aqueous  $K_2CO_3$  ( $Aldrich K_2CO_3 * \frac{3}{2} H_2O$  99+%). Electrolysis was performed with a constant current of 8 milliamps which corresponded to approximately 0.5 milliamp per square centimeter for 24 hours. The cathode was removed and rinsed with distilled water followed by XPS analysis. The spectrum in the region of 55 eV is shown as Nitest50 which is attached.

### Sample #3

This sample was the virgin nickel tubing of the gas permeation cell fabricated and tested by Thermacore, Inc. which produced 50 watts of power at 300 °C having a nickel surface area of only 300 cm<sup>2</sup>. See Exhibit 13- Shaubach, R., Gernert, N. J., "Measurement of excess heat from nascent hydrogen with potassium carbonate on nickel without electrolysis", Phys. Letts. A, in progress. The XPS spectrum performed at Lehigh University is shown in Figure 4 which is attached. The same spectrum of this sample was also obtained at National Laboratory A.

### Sample #4

This sample was the nickel tubing following the production of energy of the gas permeation cell fabricated and tested by Thermacore, Inc. The cell produced 50 watts of power at 300 °C having a nickel surface area of only 300 cm<sup>2</sup>. See Exhibit 13- Shaubach, R., Gernert, N. J., "Measurement of excess heat from nascent hydrogen with potassium carbonate on nickel without electrolysis", Phys. Letts. A, in progress. The XPS spectrum performed at Lehigh University is shown in Figure 4 which is attached. The same spectrum of this sample was also obtained at National Laboratory A.

# CHARLES EVANS & ASSOCIATES

SPECIALISTS IN MATERIALS CHARACTERIZATION

March 18, 1994

Michael Jacox  
EG&G Idaho, Inc.  
Bldg. CF-601  
Idaho National Engineering Laboratory  
Scoville, Idaho 83415

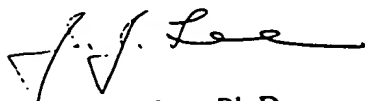
Subject: TIME-OF-FLIGHT SECONDARY ION MASS SPECTROMETRY (TOF-SIMS)  
SURFACE ANALYSIS REPORT  
CE&A Number: 40150  
Purchase Order Number:

Dear Mr. Jacox:

Enclosed is the original copy of the fax report that was sent to you. I believe that the color images in this original copy will be more useful to you

If you have any questions please do not hesitate to contact me.

Sincerely,



Jang-Jung Lee, Ph.D.  
Staff Analyst  
Organic Surface Analysis

Enclosures

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# CHARLES EVANS & ASSOCIATES

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FACSIMILE COMMUNICATION • FACSIMILE COMMUNICATION • FACSIMILE COMMUNICATION

COMPANY: EG&G Idaho Inc.

REF NO:

ATTENTION: Michael Jacox

DATE: March 18, 1994

DESTINATION

PAGE 1 OF 16

FAX NO: (208) 526-2061

CE&A REPLY FAX  
NO: (415) 369-7921

FROM: J.J. Lee

SUBJECT: TOF-SIMS Analysis

(CE&A No. 40150)

Here are the results from your Time-of-Flight Secondary Ion Mass Spectrometry (TOF-SIMS) analysis.

Purpose: To obtain mass spectra from two Ni wires, Sample # 1 and 2, and two Ni tubes, Sample # 3 and 4. Elemental contamination in the near surface layer ( $< 30$  nm) is of interest.


Experimental: TOF-SIMS mass spectra were acquired on the Charles Evans and Associates TFS system. Spectra were obtained using a gallium liquid metal ion gun (LMIG) primary ion source. The instrument was operated in an ion microprobe mode in which the pulsed primary ion beam was rastered across the sample's surface, permitting the imaging mode analysis of small features. Typical primary ion doses were on the order of  $10^{12}$  ions/cm<sup>2</sup>.

Results: The data are reported as mass spectra and ion images. Spectra are plotted as the number of secondary ions detected (Y-axis) versus the mass-to-charge ( $m/z$ ) ratio of the ions (X-axis). The ion counts are displayed on logarithmic or linear intensity scales, and probable empirical formulae for a number of the peaks are labeled on the plots. Ion images present the integrated intensities of mass selected ions within the field of view as a function of picture element, or pixel, position.

General material about the interpretation of TOF-SIMS spectra is included at the end of the report.

Discussion: The positive ion spectra of the four samples are included in Figures 1 to 10. Figures 4 to 10 contain expanded plots for various mass ranges to delineate different elemental ion peaks. In each page the spectra from the top panel are for Samples #1, #2, #3 and #4. All the spectra were acquired after a short sputter clean to remove possible surface contamination.

The spectrum of Sample #1 contains peaks for Na<sup>+</sup> (Figs 1, 4), Si<sup>+</sup> (1, 5), K<sup>+</sup> (1, 6), Ni<sup>+</sup> (1, 9), Mg<sup>+</sup> (4), Al<sup>+</sup> (5), Mn<sup>+</sup> (8) and polydimethylsiloxane (PDMS at  $m/z$  73, 147, 207, 221, etc., Figs. 1, 2 and 3). There may be very low intensity Cr<sup>+</sup> and Fe<sup>+</sup> (possible interference by Si<sub>2</sub><sup>+</sup>) in Figures 7 and 8. PDMS is a commonly found surface contamination (see attachment). The sputter clean process may have reduced the PDMS amount in the analytical area.

Sample #2 contains Na<sup>+</sup> (Figs. 1, 4), Si<sup>+</sup> (1, 5), K<sup>+</sup> (1, 6), , Mg<sup>+</sup> (4) and Al<sup>+</sup> (5). Low level Mn<sup>+</sup> ion may be present (8).

Sample #3 shows peaks for Na<sup>+</sup> (Figs. 1, 4), Si<sup>+</sup> (5), K<sup>+</sup> (1, 6), Ni<sup>+</sup> (1, 9), Mg<sup>+</sup> (4), Al<sup>+</sup> (1, 5), Pb<sup>+</sup> (3), Ca<sup>+</sup> (6), Mn<sup>+</sup> (8) Cr<sup>+</sup> (1, 7) and Fe<sup>+</sup> (1, 8).

Sample #4 contains Na<sup>+</sup> (Figs. 1, 4), Si<sup>+</sup> (5), K<sup>+</sup> (1, 6), Ni<sup>+</sup> (1, 9), Mg<sup>+</sup> (4), Al<sup>+</sup> (5), Cr<sup>+</sup> (7) and Fe<sup>+</sup> (8).

Regarding the H detection from the four samples, ion intensities for NiH<sup>+</sup> and H<sup>+</sup> are normalized to Ni<sup>+</sup> and listed in Table 1.

In summary, various elemental ion species were found in the near surface analyses of the four samples. A few ion images acquired from Samples #1, #2 and #4 are included in Images 1 to 3 respectively. It appears that the ion distributions are not uniform in the 18  $\mu$ m by 18  $\mu$ m analytical fields. The non-uniformity in distributions are clearly shown in the ion images of Ni<sup>+</sup> and K<sup>+</sup> which are the most dominant ions.

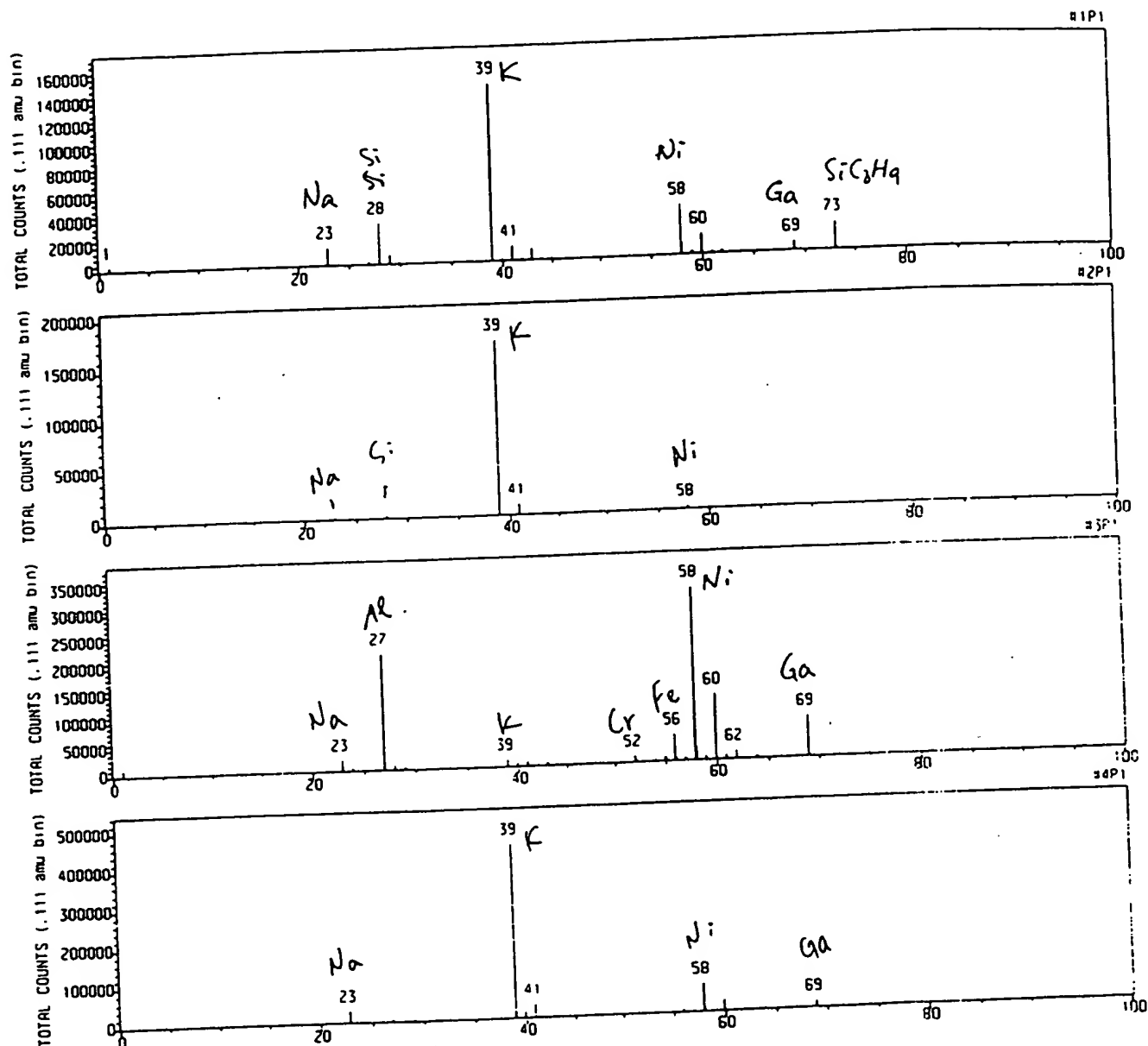
This analysis required 4 hours of instrument time at \$375.00 per hour. The total charge of \$1,500.00 will be invoiced against your purchase order. Please send us a purchase order number if you have done so. If any questions arise about these data, please feel free to contact me.

Table 1. Relative intensities of NiH<sup>+</sup> and H<sup>+</sup> (normalized to Ni<sup>+</sup>).

Samples	Ni <sup>+</sup> ion counts	H <sup>+</sup>	NiH <sup>+</sup>
#1	50230	8.2%	6.4%
#2	3331	20%	2.1%
#3	344508	3.7%	2.7%
#4	83481	4.6%	5.5%

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FILE NAME: #1P1 DATE: 15 Mar 94 13:21 ACQUISITION TIME: 21.2 MIN. TOTAL INTEGRAL 464864  
 \* IONS PRIMARY GUN: Cesium TIME RECORDER: 1-Stop TDC X-Y SOURCE: Raster TIME PER CHANNEL 15 s  
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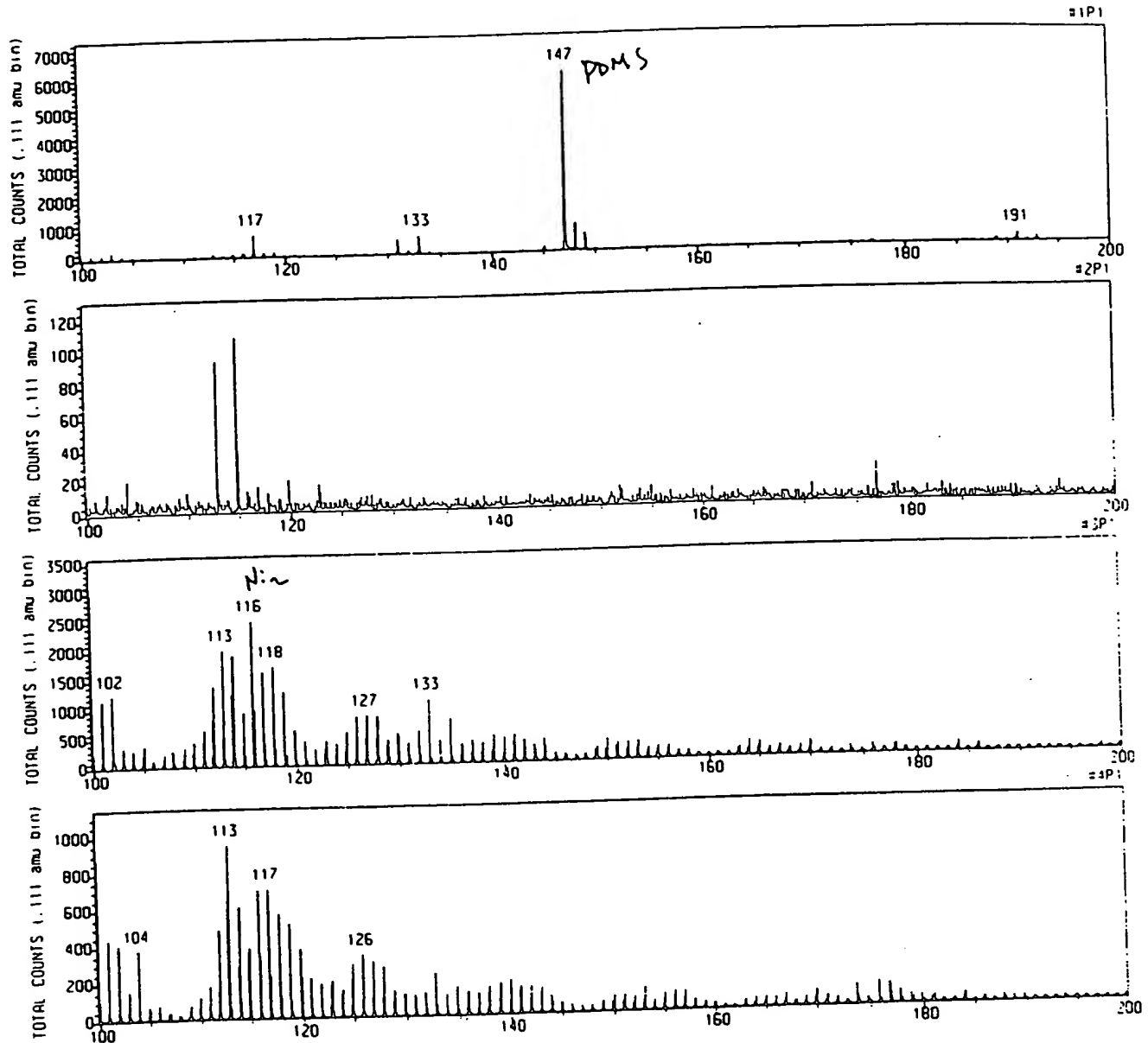
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 DATA SET: 1 Spectra: 2 Image(s) RASTER SIZE: 31µm RASTER TYPE: Full 1 4-Fold

FILE NAME: #4P1 DATE: 15 Mar 94 10:50 ACQUISITION TIME: 14.8 MIN. TOTAL INTEGRAL 824811  
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 \* IONS PRIMARY GUN: Cesium TIME RECORDER: 1-Stop TDC X-Y SOURCE: Raster TIME PER CHANNEL 15 s  
 DATA SET: 1 Spectra: 6 Image(s) RASTER SIZE: 31µm RASTER TYPE: Full 1 4-Fold

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FILE NAME: #1P1      DATE : 15 Mar 94 13:21      ACQUISITION TIME: 21.2 MIN.      TOTAL INTEGRAL 454864

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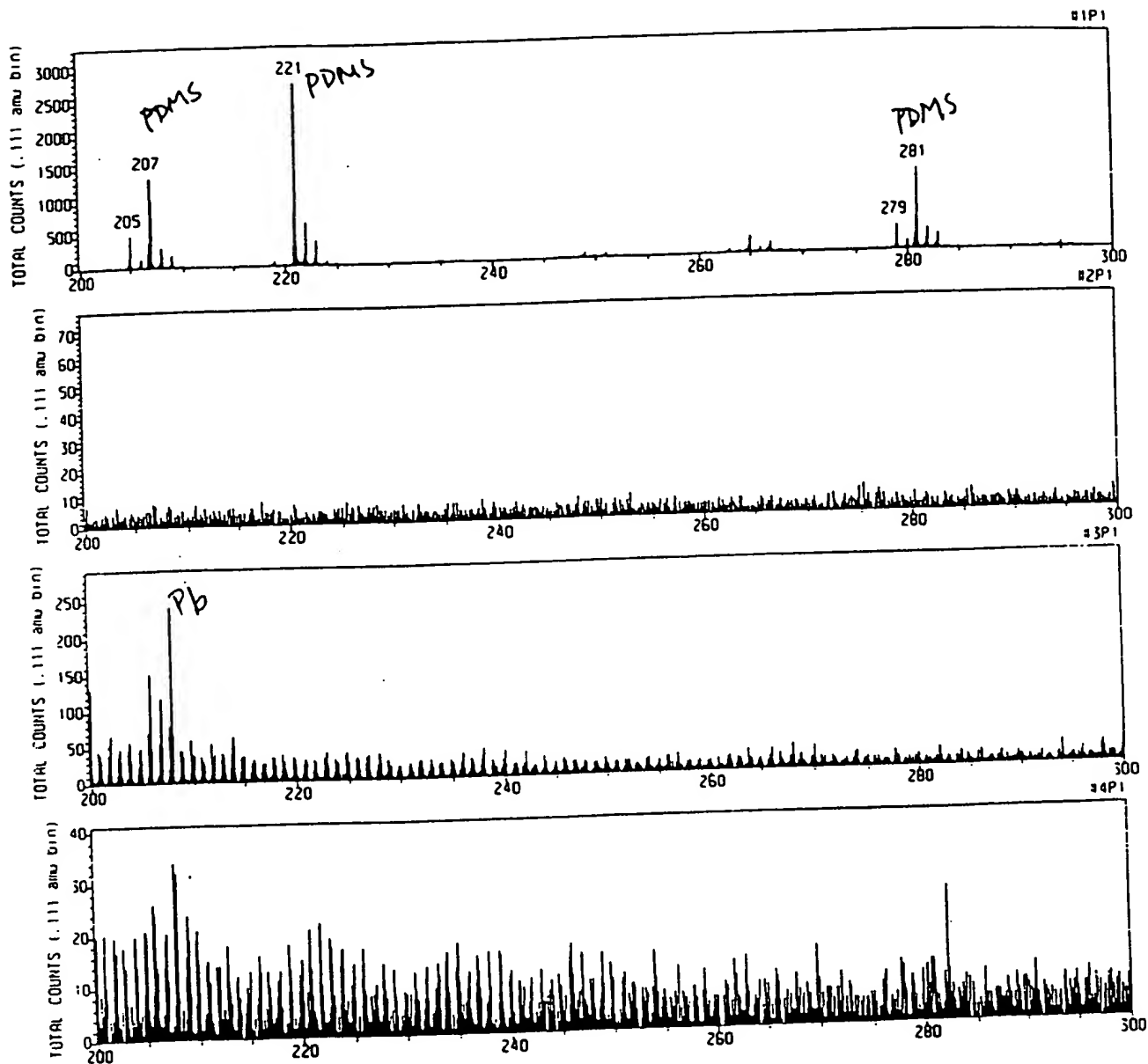
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FILE NAME: #4P1      DATE : 15 Mar 94 10:50      ACQUISITION TIME: 14.0 MIN.      TOTAL INTEGRAL : 824811

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DATA SET: 1 Spectra; 6 Image(s)      RASTER SIZE: 31µm      RASTER TYPE: Full I 4-Fold      FIGURE 1

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FILE NAME: #1P1 DATE: 15 Mar 94 13:21 ACQUISITION TIME: 21.2 MIN. TOTAL INTEGRAL: 464854

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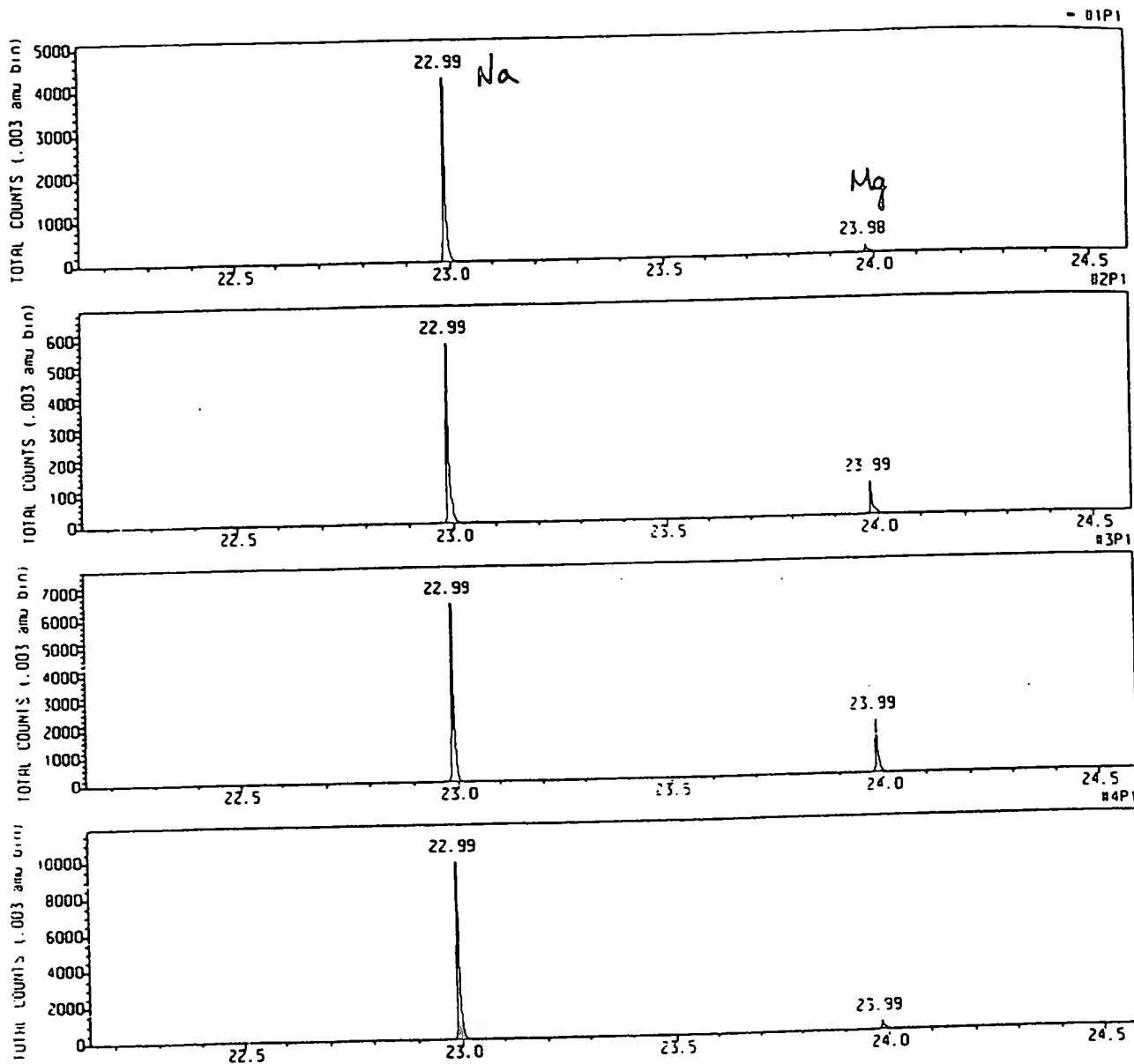
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\* IONS PRIMARY GUN: Cesium TIME RECORDER: 1-Stop TDC X-Y SOURCE: Raster TIME PER CHANNEL: 15-  
DATA SET: 1 Spectra; 2 Image(s) RASTER SIZE: 31µm RASTER TYPE: Full I 4-Fold

FILE NAME: #4P1 DATE: 15 Mar 94 10:50 ACQUISITION TIME: 14.8 MIN. TOTAL INTEGRAL: 824811

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FILE NAME #1P1 DATE : 15 Mar 94 13:21 ACQUISITION TIME: 21.2 MIN. TOTAL INTEGRAL : 464864  
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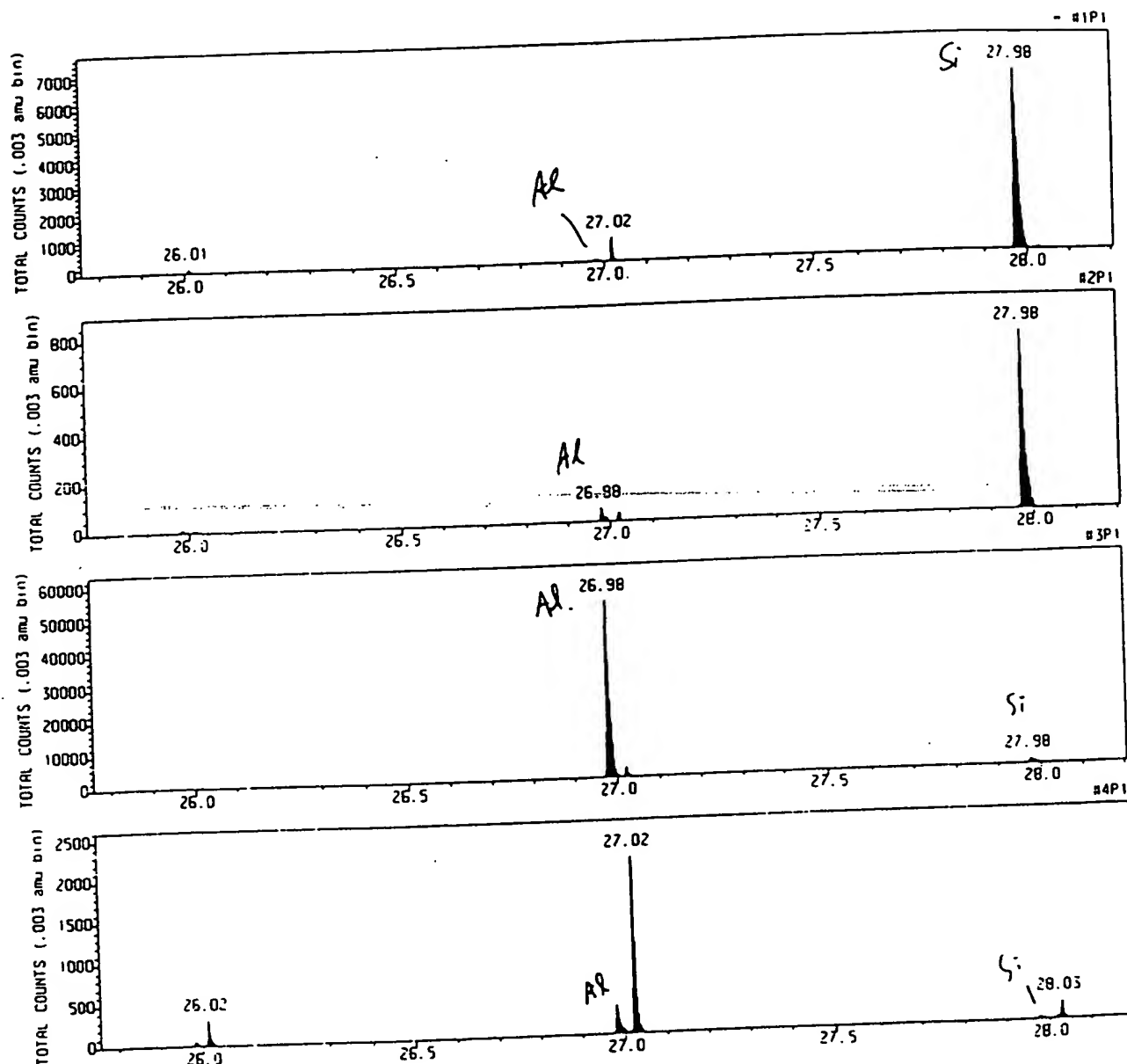
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 \* IONS PRIMARY GUN: Cesium TIME RECORDER: 1-Stop TDC X-Y SOURCE: Raster TIME PER CHANNEL: 15.4  
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FILE NAME: #4P1 DATE : 15 Mar 94 10:50 ACQUISITION TIME: 14.0 MIN. TOTAL INTEGRAL : 824811  
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 DATA SET: 1 Spectra; 6 Image(s) RASTER SIZE: 31um RASTER TYPE: Full 1 4-Fold

FIGURE —

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FILE NAME: #1P1 DATE: 15 Mar 94 13:21 ACQUISITION TIME: 21.2 MIN. TOTAL INTEGRAL: 464864  
 \* IONS PRIMARY GUN: Cesium TIME RECORDER: 1-Stop TDC X-Y SOURCE: Raster TIME PER CHANNEL: 156  
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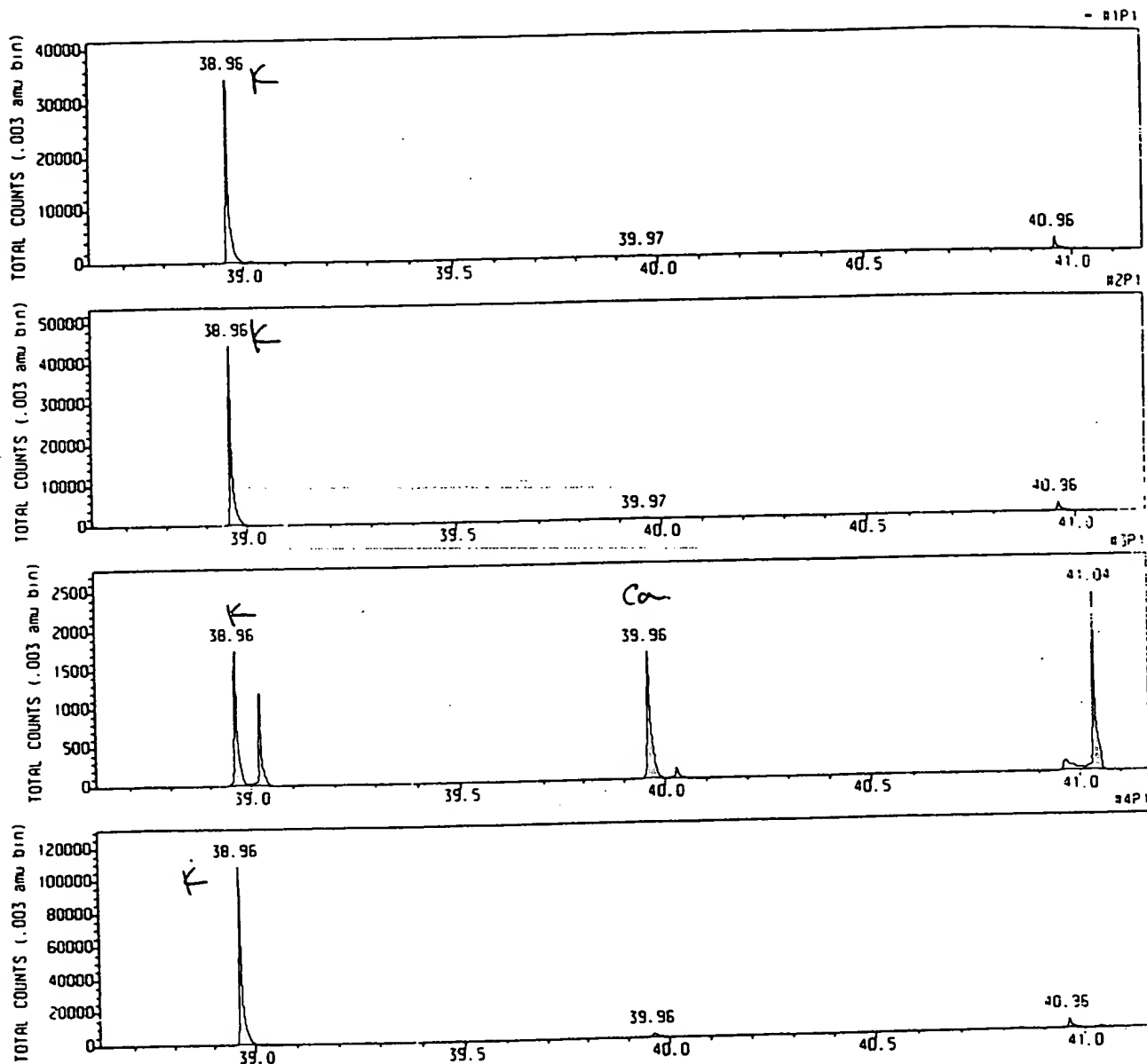
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FILE NAME: #3P1 DATE: 15 Mar 94 10:15 ACQUISITION TIME: 15.1 MIN. TOTAL INTEGRAL: 1240561  
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FILE NAME: #4P1 DATE: 15 Mar 94 10:50 ACQUISITION TIME: 14.8 MIN. TOTAL INTEGRAL: 824811  
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FIGURE —

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FILE NAME: #1P1 DATE: 15 Mar 94 13:21 ACQUISITION TIME: 21.2 MIN TOTAL INTEGRAL 454854  
+ IONS PRIMARY GUN: Cesium TIME RECORDER: 1-Stop TDC X-Y SOURCE: Raster TIME PER CHANNEL 156  
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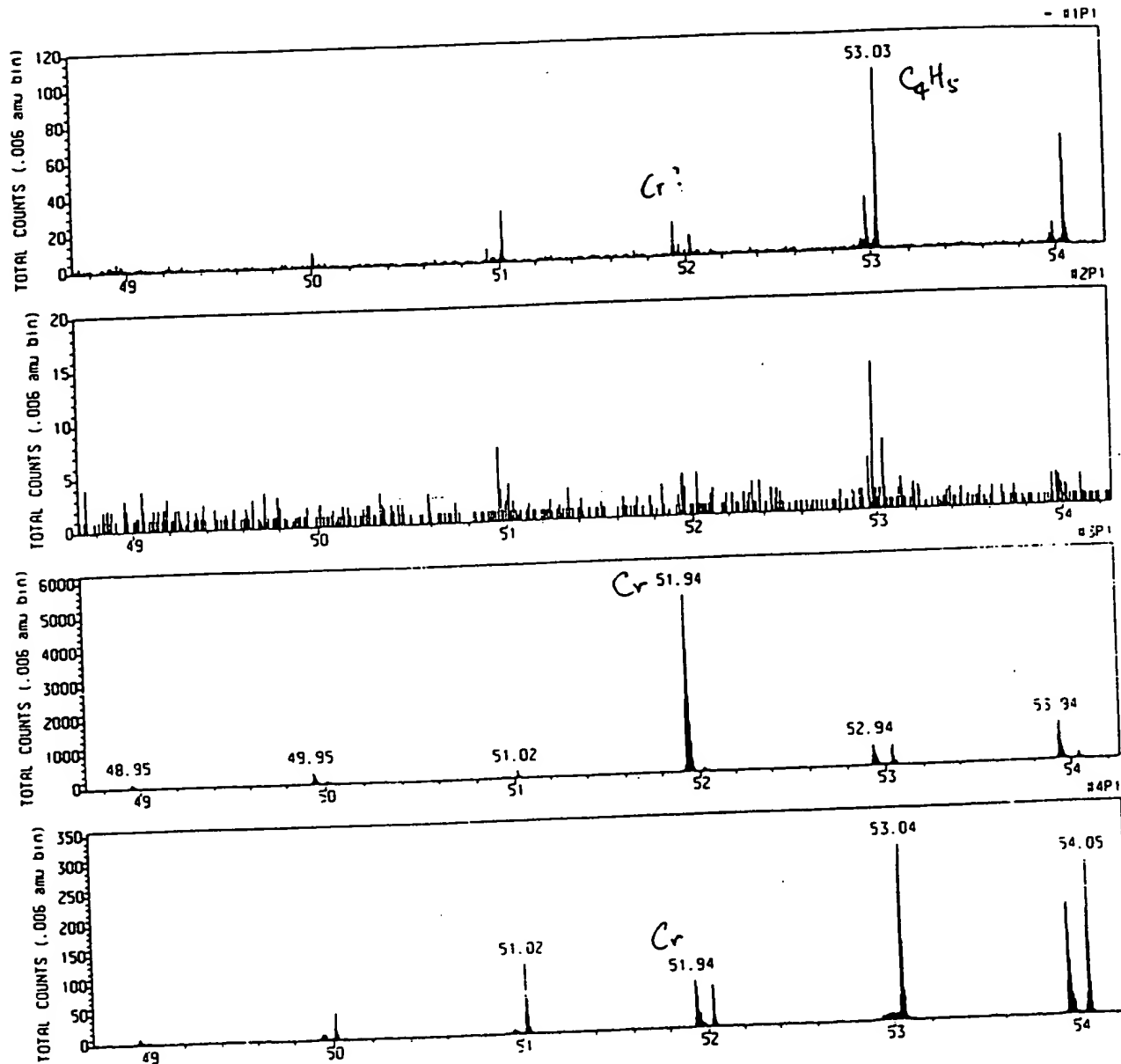
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FILE NAME: #3P1 DATE: 15 Mar 94 10:15 ACQUISITION TIME: 15.1 MIN. TOTAL INTEGRAL: 1240561  
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FILE NAME: #4P1 DATE: 15 Mar 94 10:50 ACQUISITION TIME: 14.8 MIN. TOTAL INTEGRAL: 824811  
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+ IONS PRIMARY GUN: Cesium TIME RECORDER: 1-Stop TDC X-Y SOURCE: Raster TIME PER CHANNEL 156  
DATA SET: 1 Spectra: 6 Image(s) RASTER SIZE: 31µm RASTER TYPE: Full I 4-Fold



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FILE NAME: #1P1 DATE: 15 Mar 94 13:21 ACQUISITION TIME: 21.2 MIN. TOTAL INTEGRAL: 164854  
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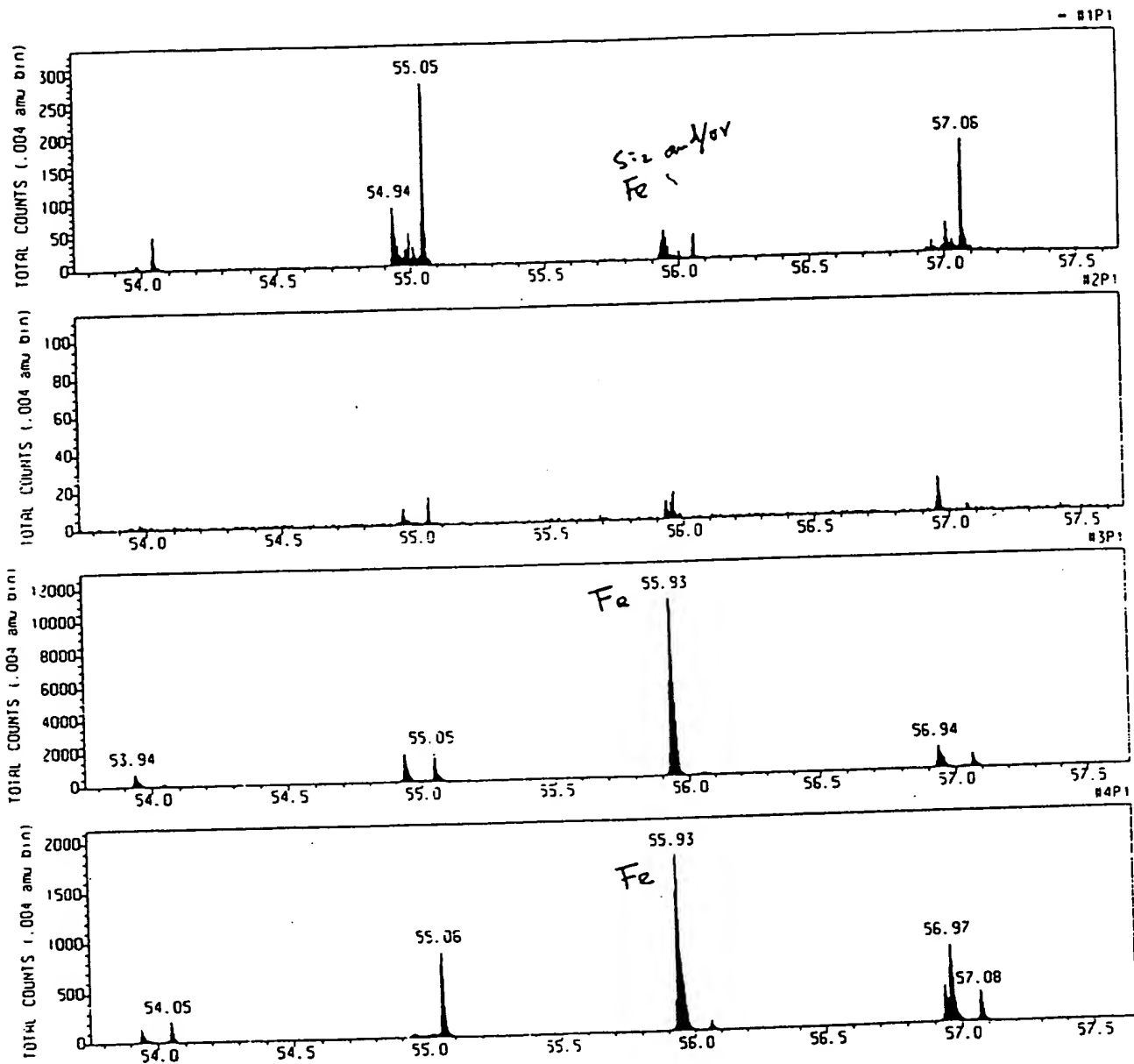
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FILE NAME: #4P1 DATE: 15 Mar 94 10:50 ACQUISITION TIME: 14.8 MIN. TOTAL INTEGRAL: 824811  
 EG&G/JACOV. #3:  
 \* IONS PRIMARY GUN: Cesium TIME RECORDER: 1-Stop TDC X-Y SOURCE: Raster TIME PER CHANNEL: 156  
 DATA SET: 1 Spectra: 6 Image(s) RASTER SIZE: 31µm RASTER TYPE: Full 1 4-Fold

FIGURE 1

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FILE NAME: #1P1 DATE: 15 Mar 94 13:21 ACQUISITION TIME: 21.2 MIN. TOTAL INTEGRAL: 454344

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\* IONS PRIMARY GUN: Cesium TIME RECORDER: 1-Stop TDC X-Y SOURCE: Raster TIME PER CHANNEL: 156  
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FILE NAME: #3P1 DATE: 15 Mar 94 10:15 ACQUISITION TIME: 15.1 MIN. TOTAL INTEGRAL: 1240561

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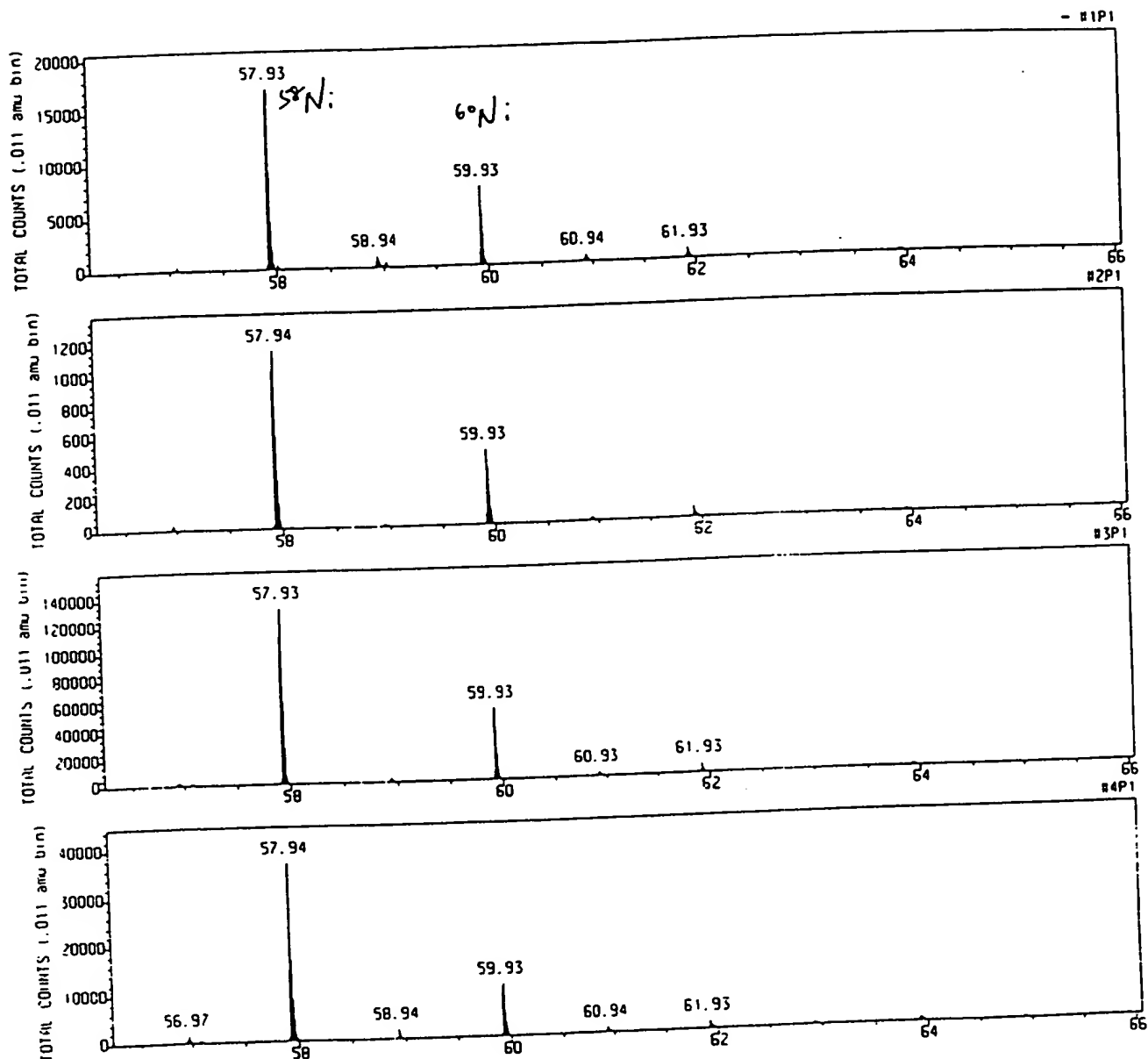
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EG&G/JACOV. #3;  
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DATA SET: 1 Spectra; 6 Image(s) RASTER SIZE: 31µm RASTER TYPE: Full I 4-Fold

FIGURE 8

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FILE NAME: #1P1 DATE: 15 Mar 94 13:21 ACQUISITION TIME: 21.2 MIN. TOTAL INTEGRAL: 464864  
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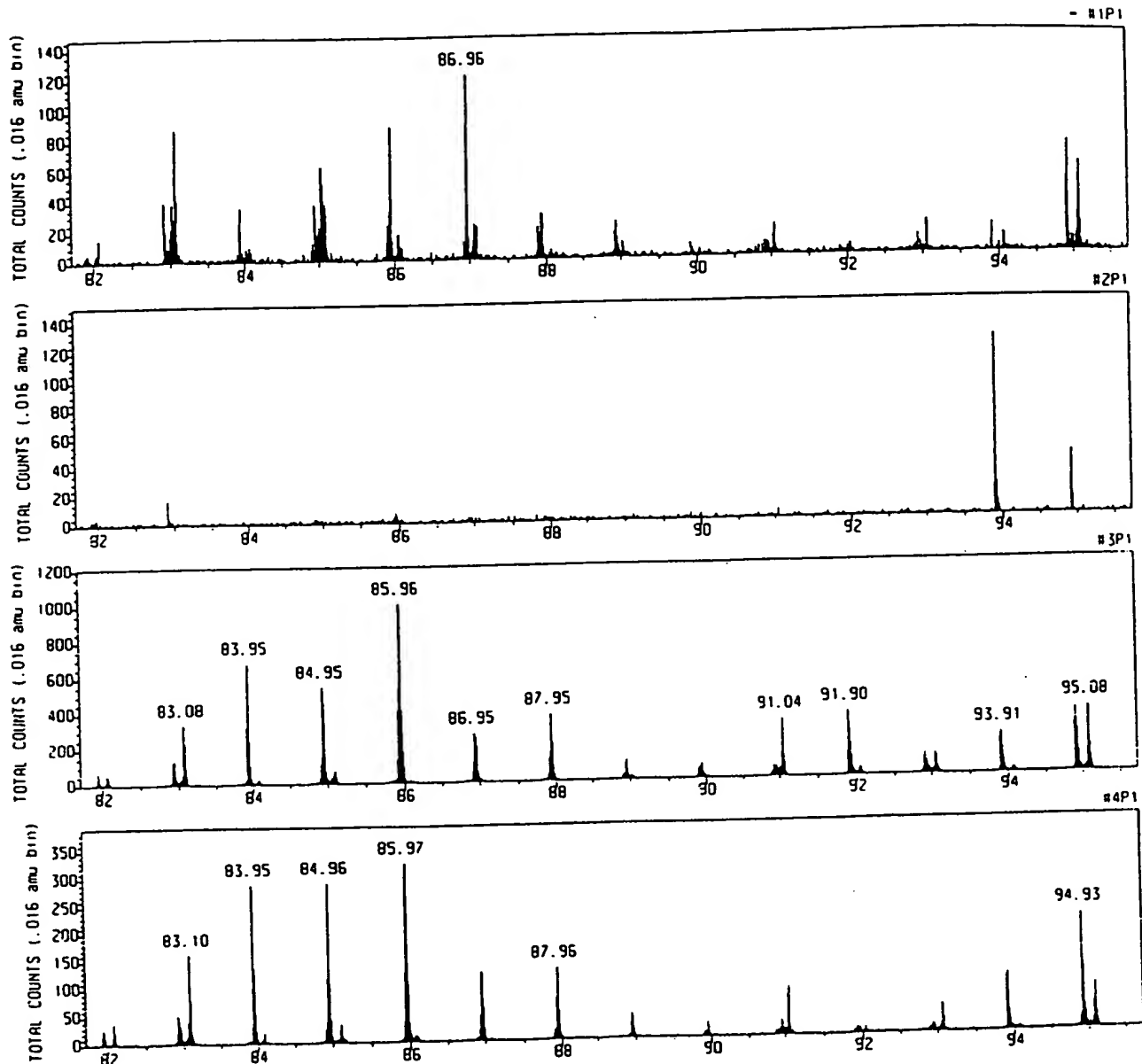
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 DATA SET: 1 Spectra: 3 Image(s) RASTER SIZE: 31µm RASTER TYPE: Full 1 4-Fold

FILE NAME: #3P1 DATE: 15 Mar 94 10:15 ACQUISITION TIME: 15.1 MIN. TOTAL INTEGRAL: 1240561  
 EG&G/JACOX, #3;  
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FILE NAME: #4P1 DATE: 15 Mar 94 10:50 ACQUISITION TIME: 14.8 MIN. TOTAL INTEGRAL: 824811  
 EG&G/JACOX, #3;  
 \* IONS PRIMARY GUN: Cesium TIME RECORDER: 1-Stop TDC X-Y SOURCE: Raster TIME PER CHANNEL: 1  
 DATA SET: 1 Spectra: 6 Image(s) RASTER SIZE: 31µm RASTER TYPE: Full 1 4-Fold

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Redwood City, CA 94063 USA  
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FILE NAME: #1P1      DATE : 15 Mar 94 13:21      ACQUISITION TIME: 21.2 MIN.      TOTAL INTEGRAL 464864

\* IONS      PRIMARY GUN: Cesium      TIME RECORDER: 1-Stop TDC      X-Y SOURCE: Raster      TIME PER CHANNEL: 15s  
DATA SET: 1 Spectra; 3 Image(s)      RASTER SIZE: 31µm      RASTER TYPE: Full I 4-Fold

FILE NAME: #2P1      DATE : 15 Mar 94 14: 6      ACQUISITION TIME: 15.0 MIN.      TOTAL INTEGRAL 230893

\* IONS      PRIMARY GUN: Cesium      TIME RECORDER: 1-Stop TDC      X-Y SOURCE: Raster      TIME PER CHANNEL 15s  
DATA SET: 1 Spectra; 3 Image(s)      RASTER SIZE: 31µm      RASTER TYPE: Full I 4-Fold

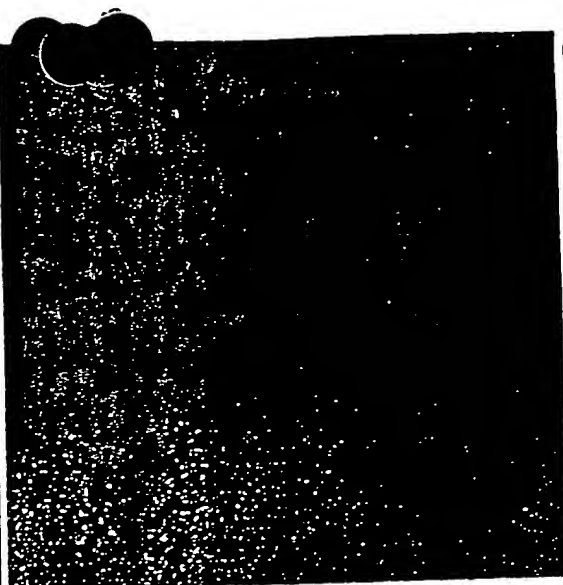
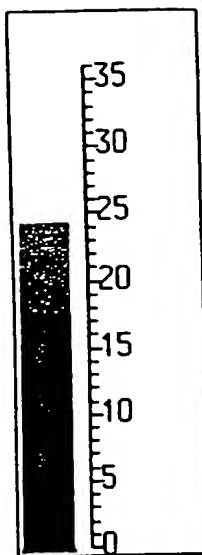
FILE NAME: #3P1      DATE : 15 Mar 94 10:15      ACQUISITION TIME: 15.1 MIN.      TOTAL INTEGRAL 1240561

EG&G/JACOX. #3:  
\* IONS      PRIMARY GUN: Cesium      TIME RECORDER: 1-Stop TDC      X-Y SOURCE: Raster      TIME PER CHANNEL: 15s  
DATA SET: 1 Spectra; 2 Image(s)      RASTER SIZE: 31µm      RASTER TYPE: Full I 4-Fold

FILE NAME: #4P1      DATE : 15 Mar 94 10:50      ACQUISITION TIME: 14.8 MIN.      TOTAL INTEGRAL : 824811

EG&G/JACOX. #3:  
\* IONS      PRIMARY GUN: Cesium      TIME RECORDER: 1-Stop TDC      X-Y SOURCE: Raster      TIME PER CHANNEL: 15s  
DATA SET: 1 Spectra; 6 Image(s)      RASTER SIZE: 31µm      RASTER TYPE: Full I 4-Fold

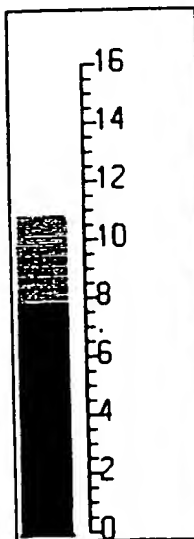
FIGURE 1



me:  
TOFIM08  
Mass:  
Lo: 0.0000  
Hi: 1000.0000  
Status:  
Saved On Disk  
Spectrum:  
#1P1  
Image Scale:

5  $\mu$ m

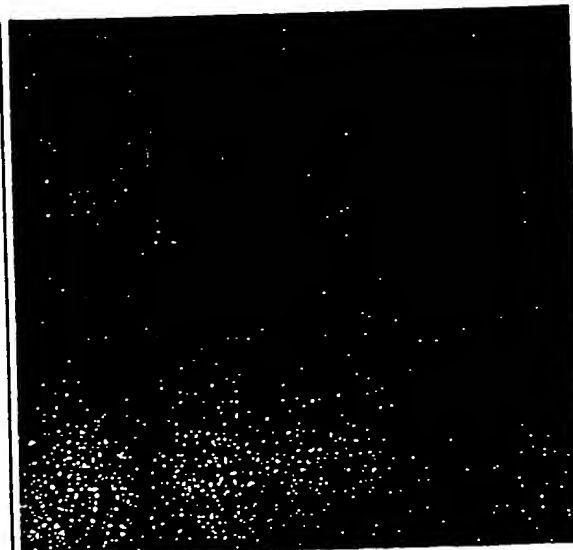
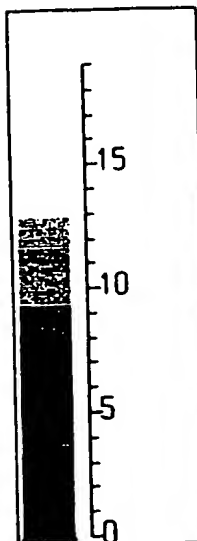
Comments: TOTAL + IONS, #1



File Name:  
TOFIM709  
Mass:  
Lo: 57.7334  
Hi: 58.1468  
Status:  
Saved On Disk  
Spectrum:  
#1P1  
Image Scale:

5  $\mu$ m

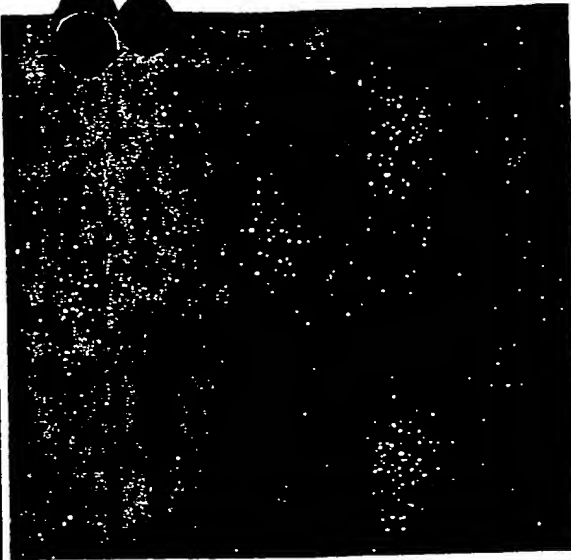
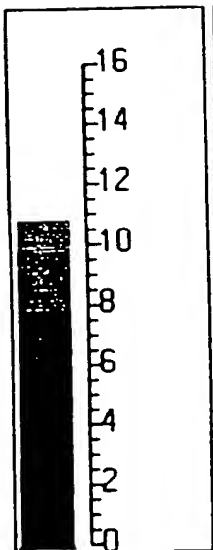
Comments: Ni+ IONS, #1



File Name:  
TOFIM710  
Mass:  
Lo: 38.7570  
Hi: 39.1704  
Status:  
Saved On Disk  
Spectrum:  
#1P1  
Image Scale:

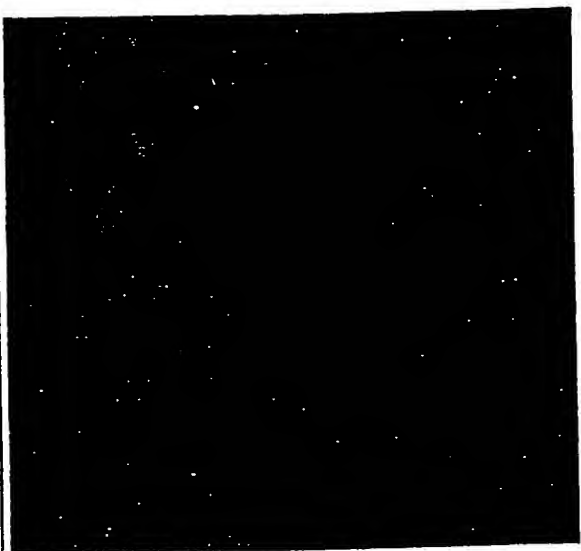
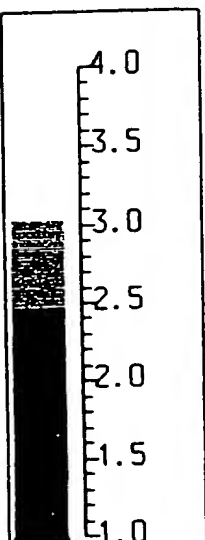
5  $\mu$ m

Comments: K+ IONS, #1



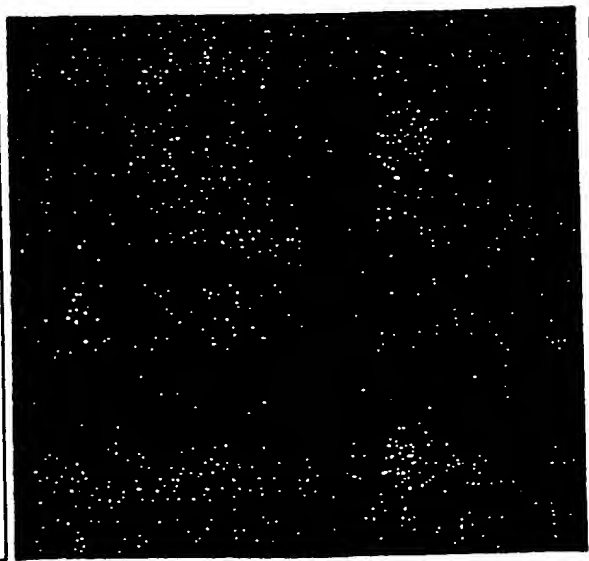
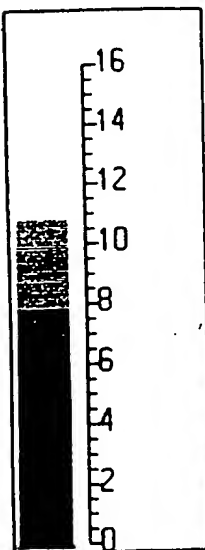
me:  
TOFIM765  
Mass:  
Lo: 0.0000  
Hi: 1000.0000  
Status:  
Saved On Disk  
Spectrum:  
#2P1  
Image Scale:  
5  $\mu$ m

Comments: TOTAL + IONS, #2



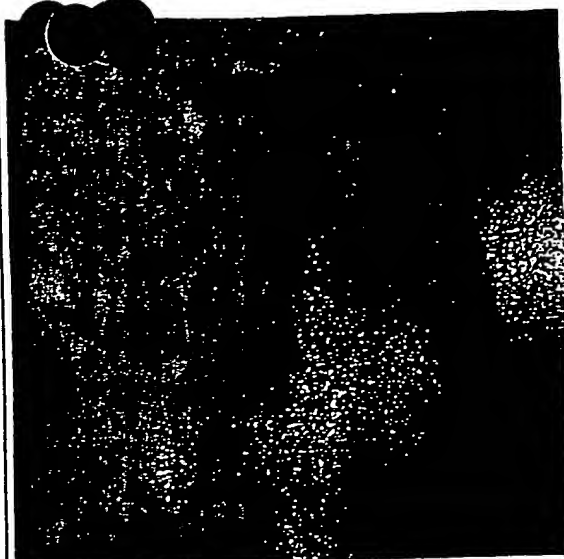
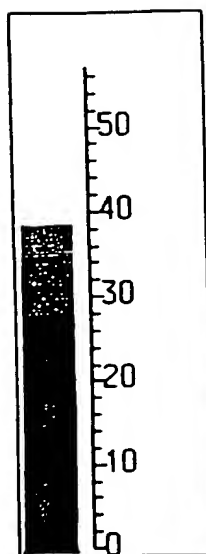
File Name:  
TOFIM766  
Mass:  
Lo: 57.7334  
Hi: 58.1468  
Status:  
Saved On Disk  
Spectrum:  
#2P1  
Image Scale:  
5  $\mu$ m

Comments: Ni+ IONS, #2



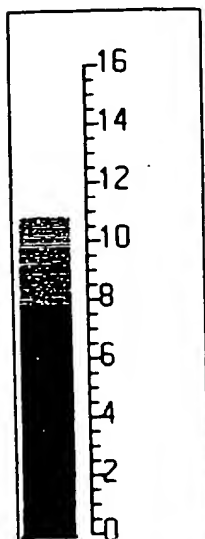
File Name:  
TOFIM767  
Mass:  
Lo: 38.7570  
Hi: 39.1704  
Status:  
Saved On Disk  
Spectrum:  
#2P1  
Image Scale:  
5  $\mu$ m

Comments: K+ IONS, #2



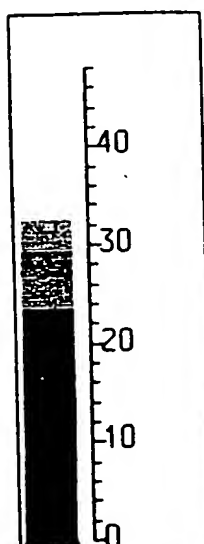
me:  
TOFIM636  
Mass:  
Lo: 0.0000  
Hi: 400.0000  
Status:  
Saved On Disk  
Spectrum:  
#4P1  
Image Scale:  
5  $\mu$ m

Comments: TOTAL + IONS, #4



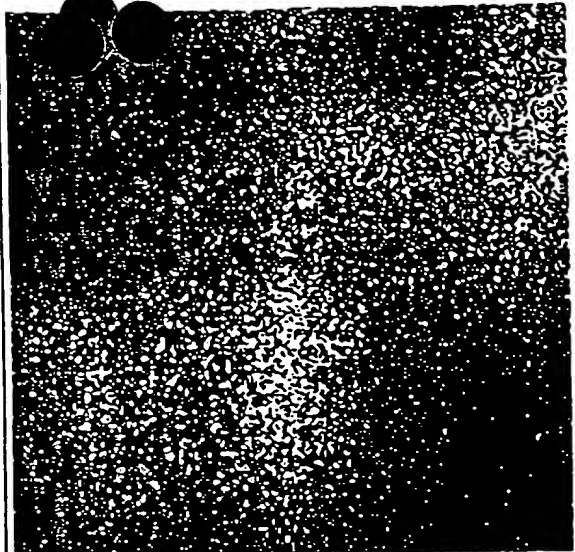
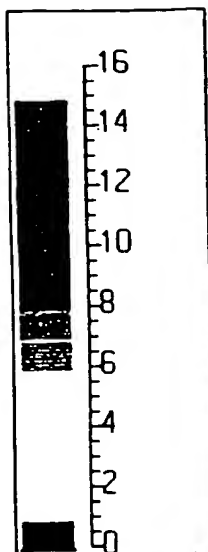
File Name:  
TOFIM637  
Mass:  
Lo: 57.7308  
Hi: 58.2193  
Status:  
Saved On Disk  
Spectrum:  
#4P1  
Image Scale:  
5  $\mu$ m

Comments: Ni+ IONS, #4



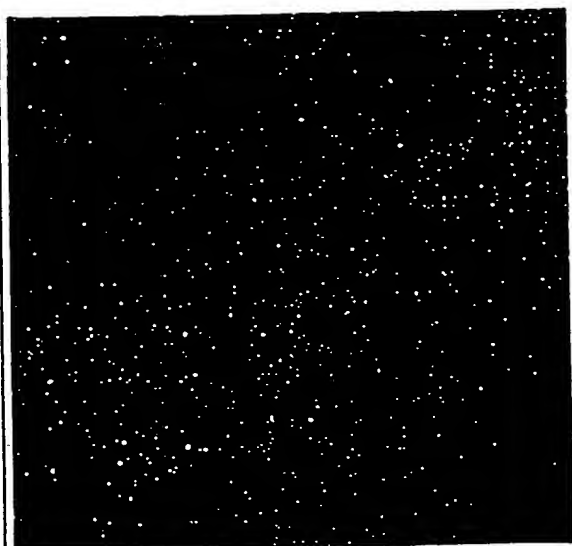
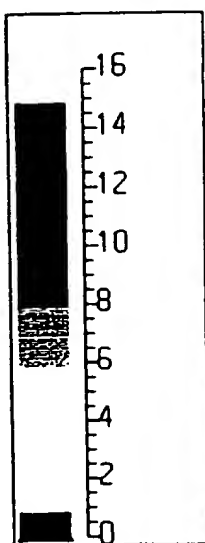
File Name:  
TOFIM638  
Mass:  
Lo: 38.9553  
Hi: 38.9730  
Status:  
Saved On Disk  
Spectrum:  
#4P1  
Image Scale:  
5  $\mu$ m

Comments: K+ IONS, #4



ame:  
TOFIM640  
Mass:  
Lo: 22.9581  
Hi: 23.0214  
Status:  
Saved On Disk  
Spectrum:  
#4P1  
Image Scale:  
5  $\mu$ m

Comments: Na+ IONS, #4

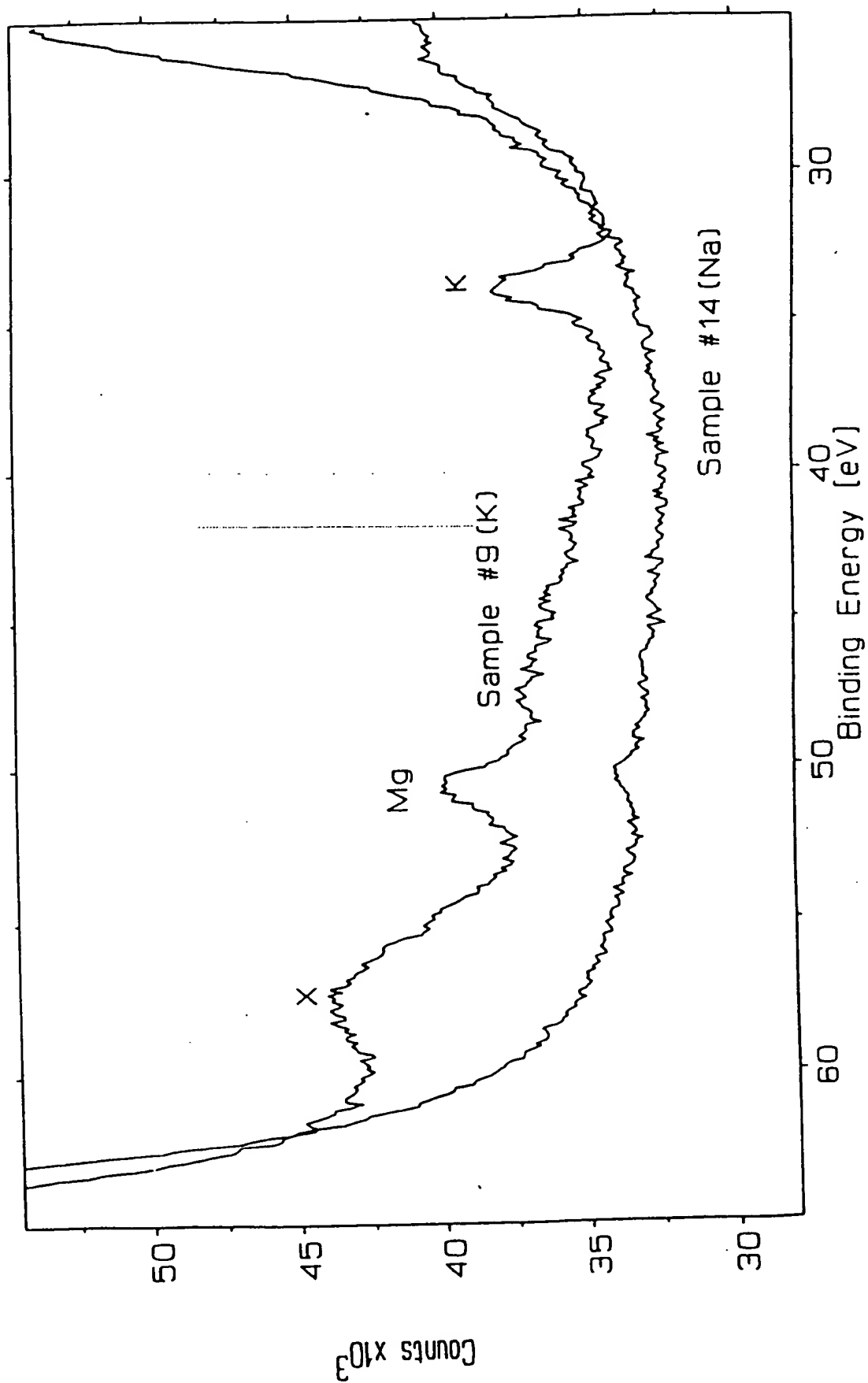


File Name:  
TOFIM641  
Mass:  
Lo: 23.9534  
Hi: 24.0166  
Status:  
Saved On Disk  
Spectrum:  
#4P1  
Image Scale:  
5  $\mu$ m

Comments: Mg+ IONS, #4

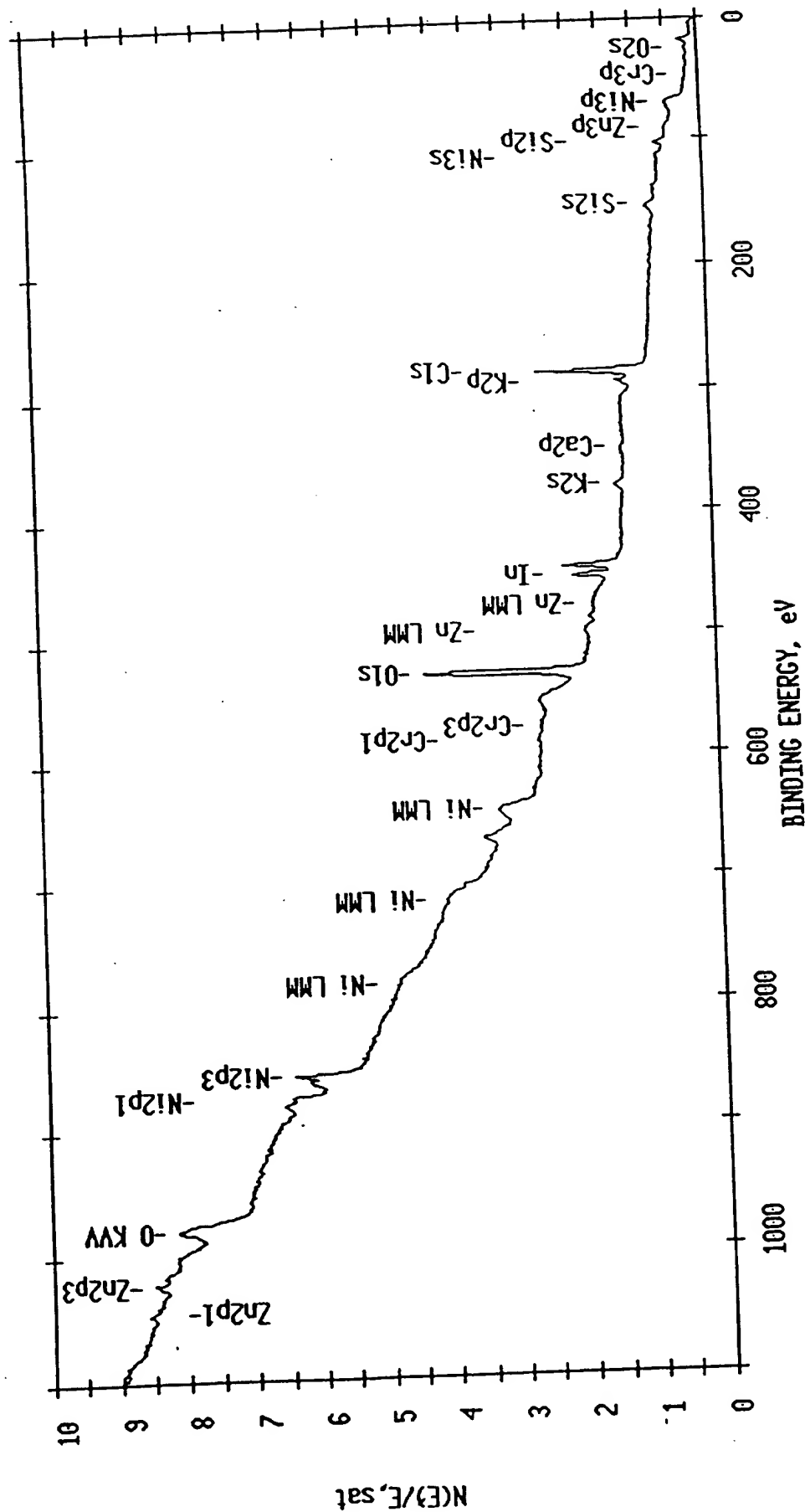


Figure 34



HPCK-09.003

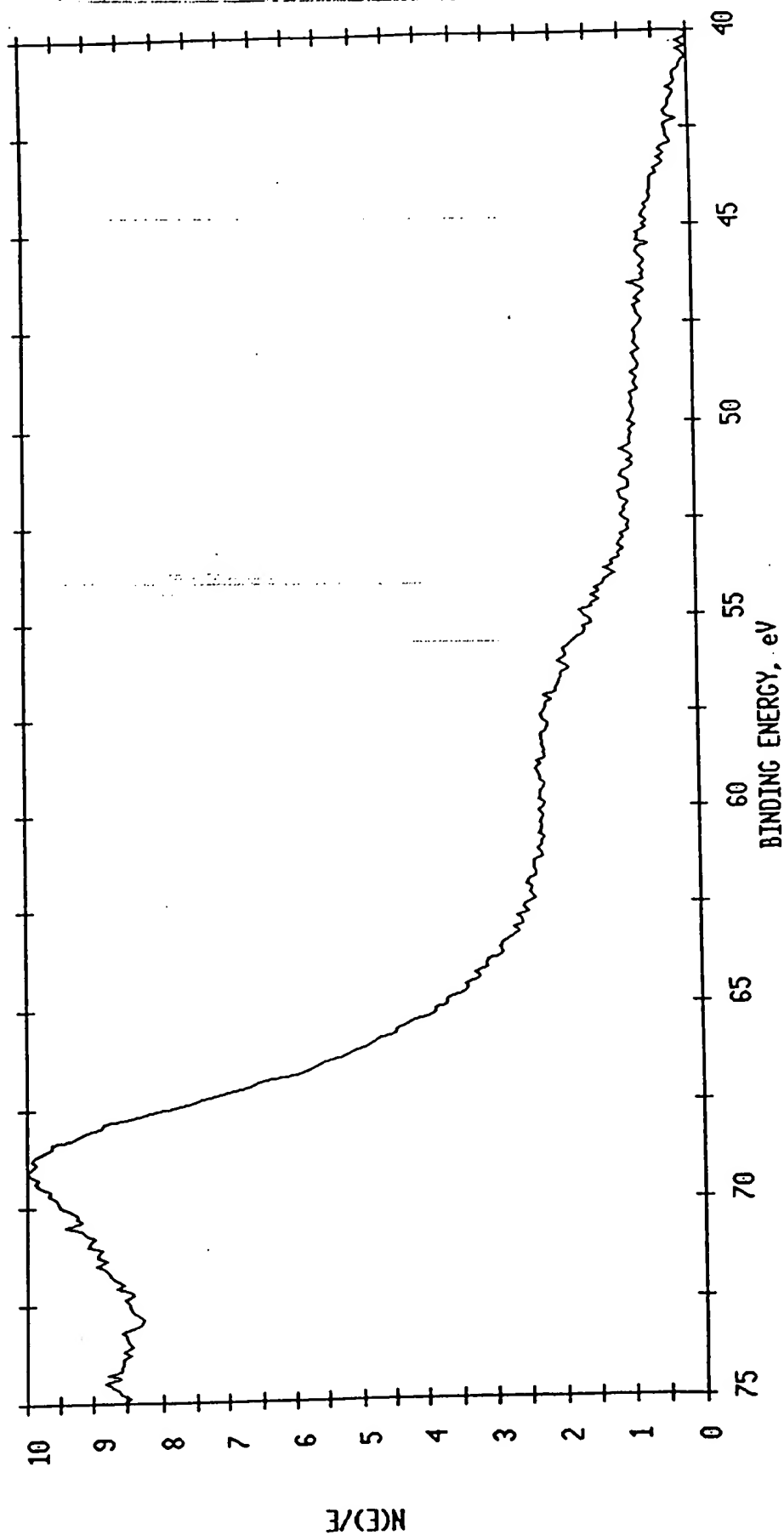
ESCA SURVEY 11/18/93 ANGLE= 15 deg ACO TIME=29.36 min  
 FILE: Nitest21 Ni wire processed in lab. as received.  
 SCALE FACTOR= 12.496 k c/s, OFFSET= 1.542 k c/s PASS ENERGY=178.950 eV Al 400 W



ESCA MULTIPLEX 11/18/93 EL= REG 2 ANGLE= 15 deg ACO TIME=67.28 min

FILE: Nitest20 Ni wire processed in lab. as received.

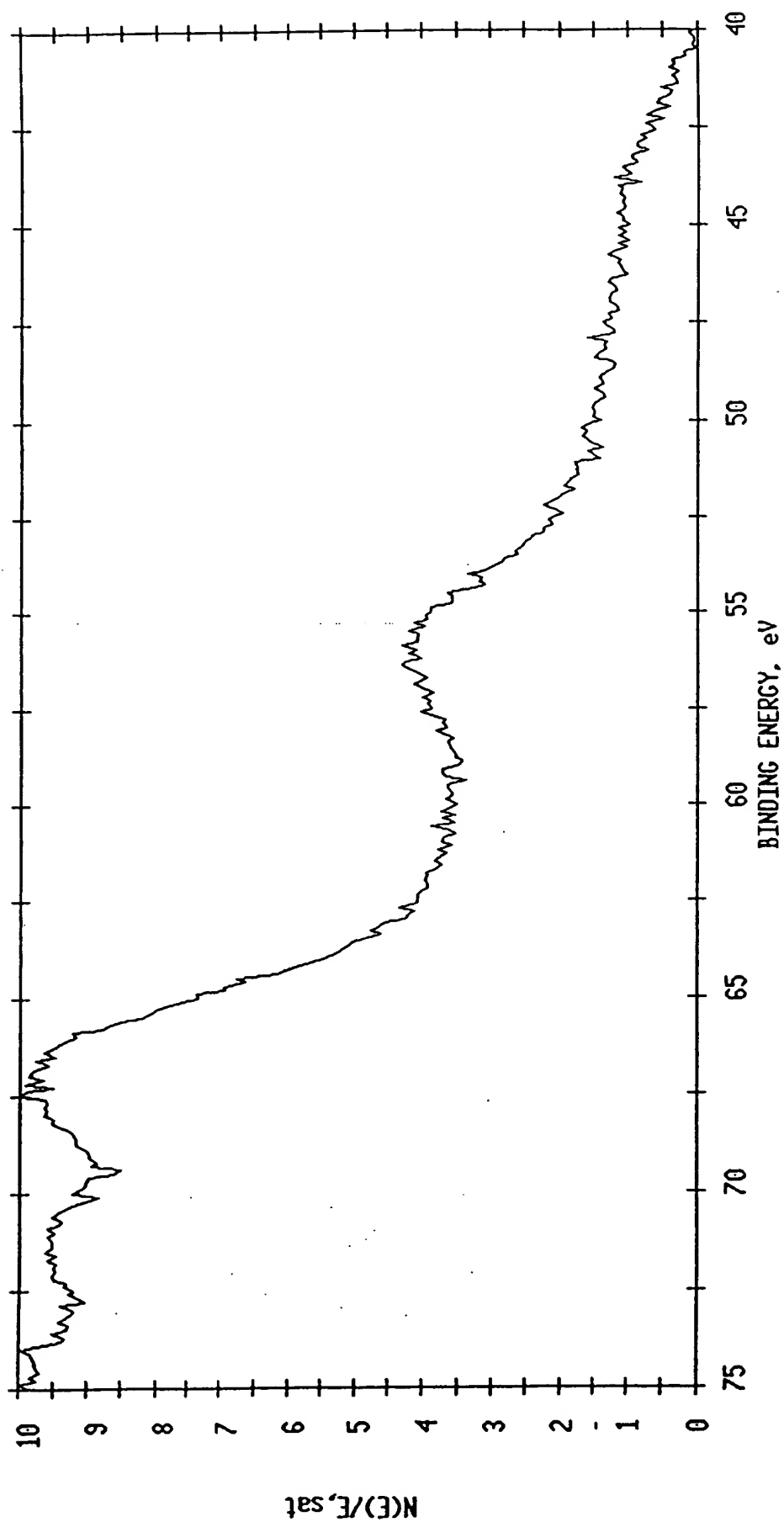
SCALE FACTOR= 0.301 k c/s, OFFSET= 2.742 k c/s PASS ENERGY=143.050 eV Al 400 W

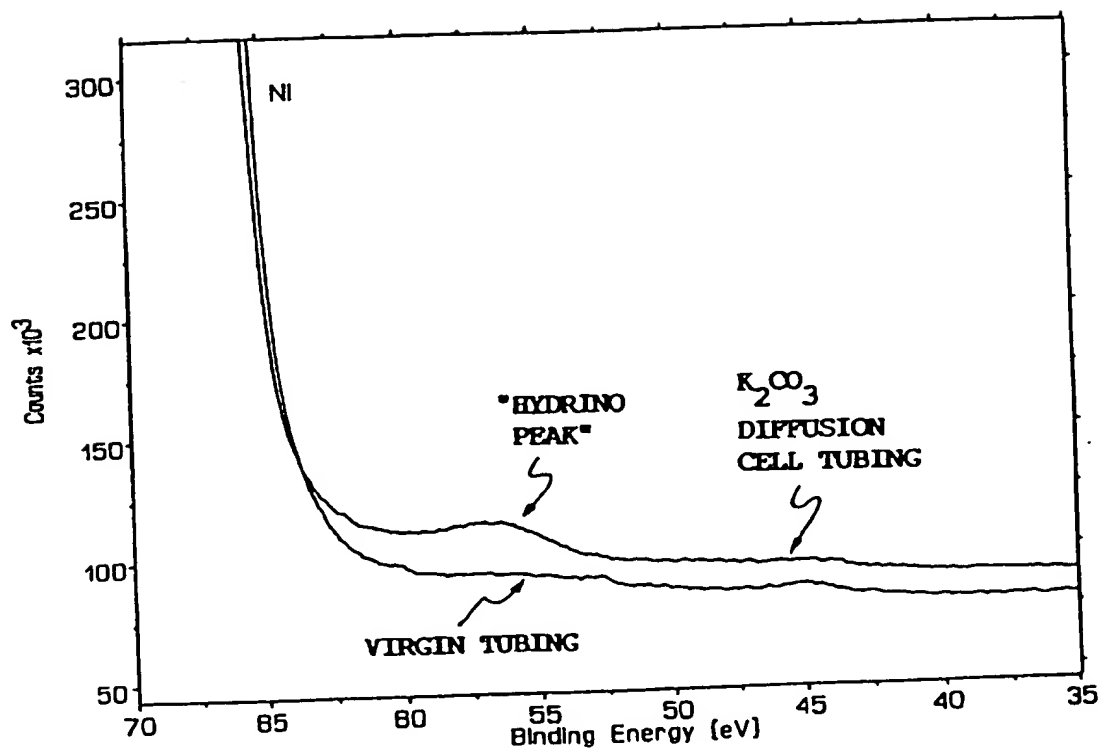


ESCA MULTIPLEX 11/24/93 EL= REG 2 ANGLE= 15 deg ACO TIME=114.08 min

FILE: Nitest50 Ni wire treated overnight at IRC.

SCALE FACTOR= 0.095 k c/s, OFFSET= 1.036 k c/s PASS ENERGY=143.050 eV AI 400 W





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